

Environmental Protection Agency

Pt. 60, App. A-8, Meth. 29

4. EPA Methods 1 through 5, *Code of Federal Regulations*, Title 40, Part 60, Appendix A, July 1, 1991.

5. EPA Method 101A, *Code of Federal Regulations*, Title 40, Part 61, Appendix B, July 1, 1991.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 29-1.—IN STACK METHOD DETECTION LIMITS (UG/M³) FOR THE FRONT-HALF, THE BACK HALF, AND THE TOTAL SAMPLING TRAIN USING ICAP, GFAAS, AND CVAAS

Metal	Front-half: probe and filter	Back-half: impinters 1-3	Back-half: impingers 4-6 ^a	Total train
Antimony	¹ 7.7 (0.7)	¹ 3.8 (0.4)	¹ 11.5 (1.1)
Arsenic	¹ 12.7 (0.3)	¹ 6.4 (0.1)	¹ 19.1 (0.4)
Barium	0.5	0.3	0.8
Beryllium	¹ 0.07 (0.05)	¹ 0.04 (0.03)	¹ 0.11 (0.08)
Cadmium	¹ 1.0 (0.02)	¹ 0.5 (0.01)	¹ 1.5 (0.03)
Chromium	¹ 1.7 (0.2)	¹ 0.8 (0.1)	¹ 2.5 (0.3)
Cobalt	¹ 1.7 (0.2)	¹ 0.8 (0.1)	¹ 2.5 (0.3)
Copper	1.4	0.7	2.1
Lead	¹ 10.1 (0.2)	¹ 5.0 (0.1)	¹ 15.1 (0.3)
Manganese	¹ 0.5 (0.2)	¹ 0.2 (0.1)	¹ 0.7 (0.3)
Mercury	² 0.06	² 0.3	² 0.2	² 0.56
Nickel	3.6	1.8	5.4
Phosphorus	18	9	27
Selenium	¹ 18 (0.5)	¹ 9 (0.3)	¹ 27 (0.8)
Silver	1.7	0.9 (0.7)	2.6
Thallium	¹ 9.6 (0.2)	¹ 4.8 (0.1)	¹ 14.4 (0.3)
Zinc	0.5	0.3	0.8

^a Mercury analysis only.

¹ Detection limit when analyzed by ICAP or GFAAS as shown in parentheses (see Section 11.1.2).

² Detection limit when analyzed by CVAAS, estimated for Back-half and Total Train. See Sections 13.2 and 11.1.3. Note: Actual method in-stack detection limits may vary from these values, as described in Section 13.3.3.

TABLE 29-2.—RECOMMENDED WAVELENGTHS FOR ICAP ANALYSIS

Analyte	Wavelength (nm)
Aluminum (Al)	308.215
Antimony (Sb)	206.833
Arsenic (As)	193.696
Barium (Ba)	455.403
Beryllium (Be)	313.042
Cadmium (Cd)	226.502
Chromium (Cr)	267.716
Cobalt (Co)	228.616
Copper (Cu)	328.754
Iron (Fe)	259.940

TABLE 29-2.—RECOMMENDED WAVELENGTHS FOR ICAP ANALYSIS—Continued

Analyte	Wavelength (nm)
Lead (Pb)	220.353
Manganese (Mn)	257.610
Nickel (Ni)	231.604
Phosphorus (P)	214.914
Selenium (Se)	196.026
Silver (Ag)	328.068
Thallium (Tl)	190.864
Zinc (Zn)	213.856

TABLE 29-3.—APPLICABLE TECHNIQUES, METHODS AND MINIMIZATION OF INTERFERENCES FOR AAS ANALYSIS

Metal	Technique	SW-846 ¹ Methods No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Fe	Aspiration	7380	248.3	Contamination	Great care taken to avoid contamination.
Pb	Aspiration	7420	283.3	217.0 nm alternate	Background correction required.
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 µl of phosphorus acid to 1 ml of prepared sample in sampler cup.
Mn	Aspiration	7460	279.5	403.1 nm alternate	Background correction required.
Ni	Aspiration	7520	232.0	352.4 nm alternate Fe, Co, and Cr	Background correction required. Matrix matching or nitrous oxide/acetylene flame
				Nonlinear response	Sample dilution or use 352.3 nm line
Se	Furnace	7740	196.0	Volatility	Spike samples and reference materials and add nickel nitrate to minimize volatilization.
				Adsorption & scatter	Background correction is required and Zeeman background correction can be useful.
Ag	Aspiration	7760	328.1	Adsorption & scatter AgCl insoluble	Background correction is required. Avoid hydrochloric acid unless silver is in solution as a chloride complex. Sample and standards monitored for aspiration rate.
Tl	Aspiration	7840	276.8		Background correction is required. Hydrochloric acid should not be used.
Tl	Furnace	7841	276.8	Hydrochloric acid or chloride	Background correction is required. Verify that losses are not occurring for volatilization by spiked samples or standard addition; Palladium is a suitable matrix modifier. 4
Zn	Aspiration	7950	213.9	High Si, Cu, & P Contamination	Strontium removes Cu and phosphate.
Sb	Aspiration	7040	217.6	1000 mg/ml Pb, Ni, Cu, or acid	Great care taken to avoid contamination.
					Use secondary wavelength of 231.1 nm; match sample & standards acid concentration or use nitrous oxide/acetylene flame.
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction.
As	Furnace	7060	193.7	Arsenic Volatilization Aluminum	Spike samples and add nickel nitrate solution to digestates prior to analysis. Use Zeeman background correction.
Ba	Aspiration	7080	553.6	Calcium.	
				Barium Ionization	High hollow cathode current and narrow band set.
Be	Aspiration	7090	234.9	500 ppm Al. High Mg and Si	2 ml of KCl per 100 ml of sample.
Be	Furnace	7091	234.9	Be in optical path	Add 0.1% fluoride.
Cd	Aspiration	7130	228.8	Absorption and light scattering	Optimize parameters to minimize effects.
Cd	Furnace	7131	228.8	As above	Background correction is required.
				Excess Chloride	As above.
					Ammonium phosphate used as a matrix modifier.
					Use cadmium-free tips.
Cr	Aspiration	7190	357.9	Pipet Tips	
				Alkali metal	KCl ionization suppressant in samples and standards—Consult mfgs' literature.
Co	Furnace	7201	240.7	Excess chloride	Use Method of Standard Additions.
Cr	Furnace	7191	357.9	200 mg/L Ca and P	All calcium nitrate for a known constant effect and to eliminate effect of phosphate.

Cu	Aspiration	7210	324.7	Absorption and Scatter	Consult manufacturer's manual.
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¹ Refer to EPA publication SW-846 (Reference 2 in Section 16.0).